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## REJUVENATION OF ASPHALT PAVEMENTS

F. S. Rostler

R. M. White

Materials Research and Development, Inc.

TECHNICAL REPORT NO. AFWL-TR-70-83

December 1970

AIR FORCE WEAPONS LABORATORY

Air Force Systems Command

Kirtland Air Force Base

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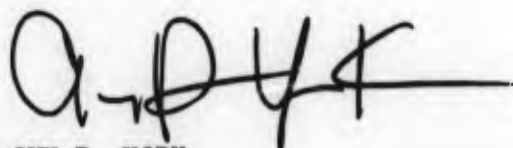
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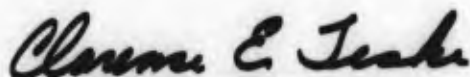
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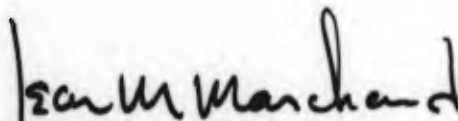
This technical report has been reviewed and is approved.



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## ABSTRACT

(Distribution Limitation Statement No. 3)

The physical and chemical aspects of the manufacture of paving grade asphalts and of asphalt performance are presented in sufficient detail to provide background information for understanding the physical, chemical and mechanical aspects of paving rejuvenation. The principal facts discussed are substantiated by references to the technical literature, which should be consulted for additional information regarding details. Theoretical and practical considerations pertaining to the use of asphalt rejuvenators are discussed, and laboratory methods of evaluating the utility of asphalt rejuvenators are detailed. The present status of knowledge regarding asphalt manufacture, causes of deterioration of asphalt pavements, and reversibility of the deterioration are depicted in graphs and flow charts. Five specimens, typical of commercially available asphalt rejuvenators, and tests performed on them are described. The test results obtained are tabulated. It is recommended that field tests be conducted before establishing standardized laboratory procedures for acceptance specifications for asphalt rejuvenators. Criteria to be used in tentative specifications to be issued before field testing are suggested.

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## SECTION I

### INTRODUCTION

The objectives of the study were to determine causes of asphalt pavement deterioration, to establish desirable characteristics of materials that can be applied to an asphalt surface for the purpose of rejuvenating it, and to provide background information needed for preparation of a performance specification for pavement rejuvenation.

The report has been organized to cover the following topics:

1. The chemistry and production of paving grade bitumen.
2. Causes of deterioration of asphalt surfacing and reversibility of the deterioration.
3. Restoration of properties of aged asphalt by addition of asphalt components.
4. Commercial asphalt rejuvenators.
5. Laboratory tests of asphalt rejuvenators and interpretation of results.
6. Criteria for specifications for asphalt rejuvenators, and suggested tentative limits.
7. Suggested field tests of rejuvenators.

Test methods used are described in appendices. Identification of the proprietary products by trade names and company releases are in the files of the contracting agency together with copies of correspondence with manufacturers and users.

## SECTION II

### SUMMARY REVIEW OF PUBLISHED TECHNICAL INFORMATION ON ASPHALT MANUFACTURE AND PERFORMANCE

#### 1. THE PRODUCTION OF PAVING GRADE BITUMENS AND THEIR BASIC PROPERTIES

The purpose of this review of origin and manufacture of paving grade bitumens is to summarize the basic information needed to understand the nature of bituminous cements and their inherent properties which have a bearing on performance in a pavement. Comprehensive textbooks deal with the chemical and physical properties of petroleum products. Among the most authoritative are Science of Petroleum (Ref. 1) and Scientific Fundamentals of Petroleum Processing (Ref. 2).

Nearly all paving grade bitumens used are produced from petroleum. Other products are coal tar bitumens and natural asphalts such as gilsonite and Trinidad asphalt. The term "petroleum bitumen" embraces crude petroleum oil and all materials derived from crude oil. The nomenclature employed to describe bitumens varies considerably, particularly as used in different countries. The term "asphalt bitumen" is, however, almost exclusively reserved for petroleum asphalt, which is the product of primary interest in this project.

Petroleum asphalt comprises a mixture of nondistillable compounds and relatively high-boiling compounds of petroleum or of other natural bitumens related to petroleum. The principal definitive requirement of all asphalt bitumens is solubility in carbon disulfide. Petroleum asphalt is a mixture of a multitude of chemical compounds which cannot be analytically separated or quantitatively determined as individuals. In realization of this fact, a number of methods have been suggested to analyze and

define asphaltic bitumens by selected physical properties or chemical behavior believed to be typical and significant for individual asphalts. A critical review of these methods has been published previously (Ref. 3). One of the chemical methods of asphalt analysis, the acid precipitation method, defines asphalts as consisting of groups of components differing in their chemical reactivity (Refs. 4, 5, 6). It has been shown in extensive investigations (Refs. 5, 6, 7, 8, 9, 10) that composition in terms of this analysis governs the behavior of asphalts, particularly in aging.

The ultimate analysis of asphalt as determined by combustion methods shows that carbon and hydrogen constitute usually more than 90 percent of its elementary composition. Other elements contained in nearly all asphalts are nitrogen, sulfur, and oxygen. Traces of various metals are also present in asphalts. The basic chemical makeup of asphalts is that of hydrocarbons and hydrocarbon derivatives, which can be considered oxidation products of the hydrocarbons and compounds in which sulfur or nitrogen atoms have taken the place of some of the carbon atoms.

For the sake of uniformity in explaining the phenomena which are of consequence in the present study and because the precipitation method of asphalt analysis is considered by the contractor to be the most reliable and informative analytical procedure, this method has been used throughout this study.

Tables I and II and figures 1 through 5 are presented to review and summarize the chemical and physical characteristics of petroleum bitumens pertinent to this project.

Table I presents as a general orientation, a classification of hydrocarbons and their over-all characteristics.

In addition to chemical definition of asphalts, physical characteristics are also used to define and identify individual asphalts and asphalt grades. One of the principal physical characteristics is consistency, which is usually numerically determined as resistance to penetration of a standardized needle into asphalt under specified conditions of load, temperature

Table I

## HYDROCARBON TYPES AND TYPICAL CHARACTERISTICS



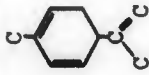
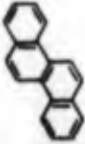

Hydrocarbon Type	Paraffinic hydrocarbons	Isoparaffins and paraffins of mixed structure	Cycloparaffins (naphthenes)	Olefins, cyclo-olefins, and terpenes and other cyclo-olefins with branched side chains	Polynuclear aromatic hydrocarbons	Aromatic hydrocarbons
Typical structures	C-C-C-C-C	$\begin{array}{c} \text{C}-\text{C}-\text{C}-\text{C} \\   \\ \text{C} \end{array}$ 				
	low-octane gasoline	high-octane gasoline	petroleum solvents	turpentine	coal tar and residues from catalytic cracking of petroleum	coal tar
Typical products associated with type as to chemical nature	paraffin wax	lubricating oils	medicinal white oil	pine tar	nitration products alkali-insoluble	inert, high solvent power
	Low boiling	High boiling				
Typical chemical character	inert	inert	inert, high solvent power	subject to auto-oxidation and resinification	partially soluble	partially soluble
Behavior towards cold concentrated $\text{H}_2\text{SO}_4$	insoluble	insoluble	insoluble	soluble, sludge-forming		

Table II

## CHARACTERISTICS OF FRACTIONAL COMPONENTS OF ASPHALTS

Fraction	General Description	Specific Chemical Reactivity	TYPICAL ANALYTICAL DATA								Significant Function
			Elementary Composition, %					$n_D^{25}$	$d_4^{20}$	I No.	
			C	H	N	S	O				
A Asphaltenes	Higher Molecular weight condensation products	Insoluble in n-pentane	86.5	8.3	2.3	1.5	1.4	--	--	--	Bodying agent
N Nitrogen Bases	Petroleum resins containing nitrogen bases and other highly reactive compounds	Precipitable with 85% $H_2SO_4$	86.1	9.3	3.0	0.7	0.9	1.642	1.08	--	Peptizer for asphaltenes
A <sub>1</sub> First Acidaffins	Resinous hydrocarbons	Precipitable with concentrated $H_2SO_4$	90.0	7.8	0.3	1.9	--	1.613	1.07	65-100	Solvent for peptized asphaltenes
A <sub>2</sub> Second Acidaffins	Slightly unsaturated hydrocarbons	Precipitable with fuming $H_2SO_4$ (30% $SO_3$ )	88.8	9.6	0.0	0.9	0.7	1.558	1.01	5	Solvent for peptized asphaltenes
P Paraffins	Saturated hydrocarbons	Non-reactive with fuming $H_2SO_4$ (30% $SO_3$ )	86.5	13.5	--	--	--	1.486	0.90	--	Gelling agent



and time (ASTM Procedure D5) or as resistance to flow. The penetration test is also considered a hardness test. Resistance to flow is expressed numerically as viscosity under specific conditions of temperature, shear and velocity of flow. Viscosity is considered a fundamental property and is often expressed in terms of fundamental units (ASTM Procedures D2170, D2171).

Figures 1 through 5 and table II are reproduced here from the quoted published literature to illustrate the basic facts summarized above. Figure 1 is a general diagram of subdivisions of bitumen. Figure 2 is a flow chart depicting manufacture of various asphalt products. Figure 3 is a flow chart of the acid precipitation method. Figures 4 and 5 are flow diagrams of a typical petroleum refining operation in terms of the acid precipitation method and in terms of elementary composition, respectively. Table II gives the characteristics of fractional components of asphalts.

The first step in the manufacture of asphalt is its separation from the crude oil. This step is, in the majority of manufacturing procedures, distillation, which yields asphalt as residue. Propane precipitation is another method of separating the asphaltic residue from the crude oil.

The distillation process separates crude oil into asphalt and distillates. The distillates are then further cut by solvent extraction (refining) into resins (extracts) and oils (raffinates). Figure 4 depicts a typical distribution of the components resulting from the manufacturing process in terms of the fractional components determined and defined by the analysis shown in figure 3.

The consistency of the asphalt obtained depends on how much has been distilled over. The various penetration grades (or viscosity grades) are produced by distilling to the desired consistency of the residue, or by fluxing a harder residue with distillates, extracts, raffinates or blends of those fluxes. Soft asphaltic base material obtained by precipitation with propane or from the distillation of some crudes is usually brought into grade, i.e., hardened by air-blowing. Hard and soft asphalts may be blended to the desired consistency.



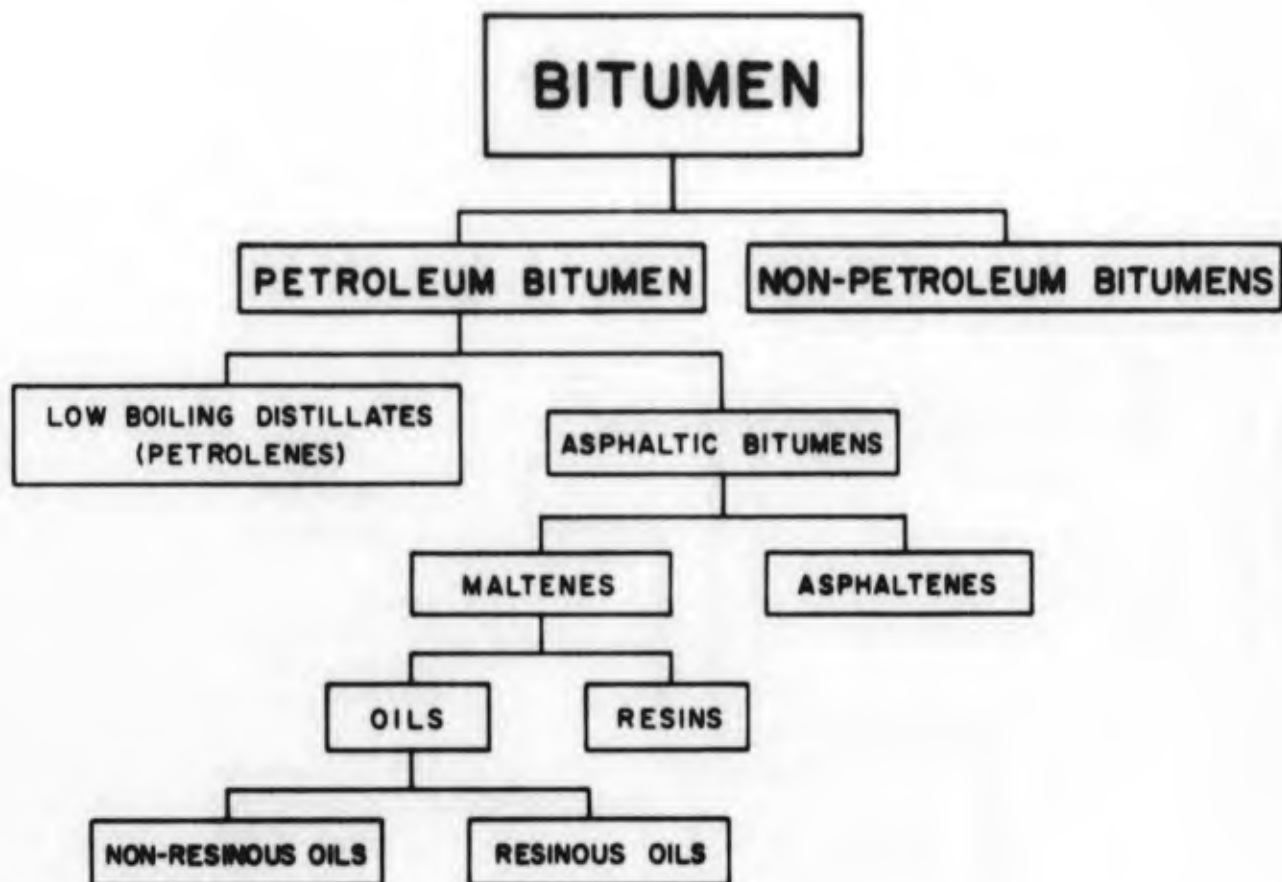


Figure 1. General Diagram of Subdivisions of Bitumen

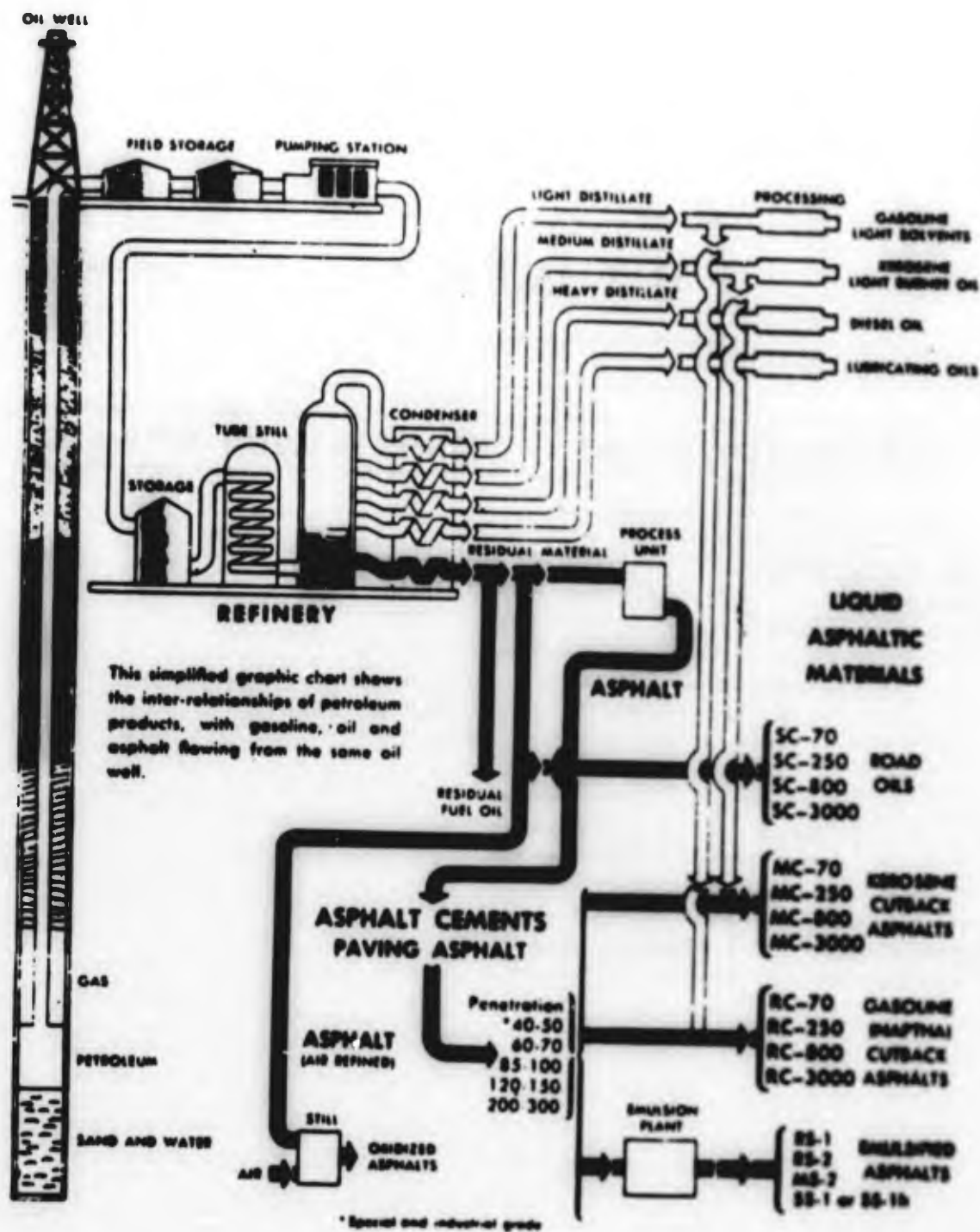


Figure 2. Petroleum Asphalt Flow Chart  
(Reproduced from The Asphalt Handbook  
by permission of The Asphalt Institute)

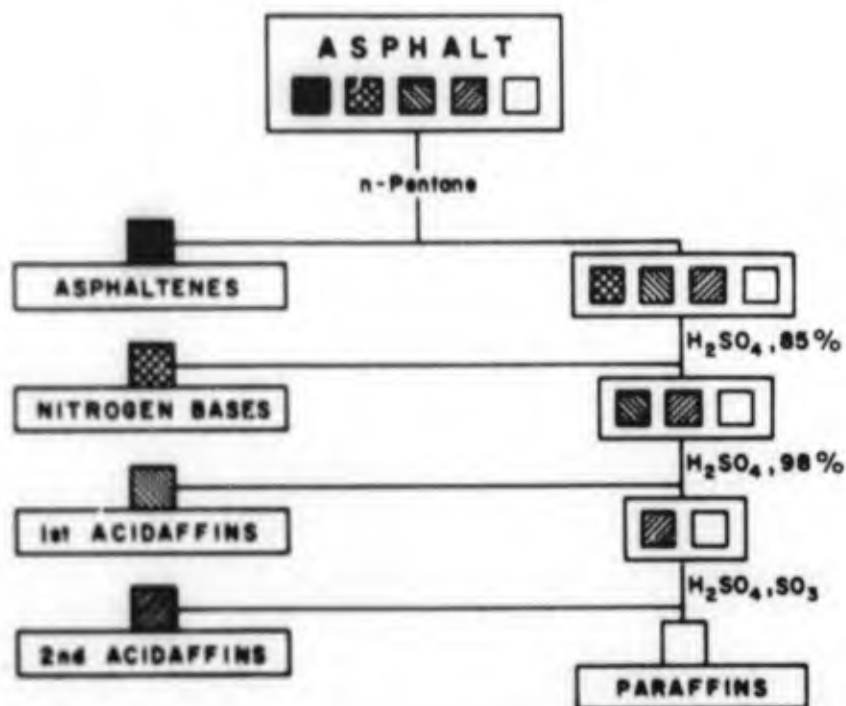


Figure 3. Flow Sheet of Acid Precipitation Method

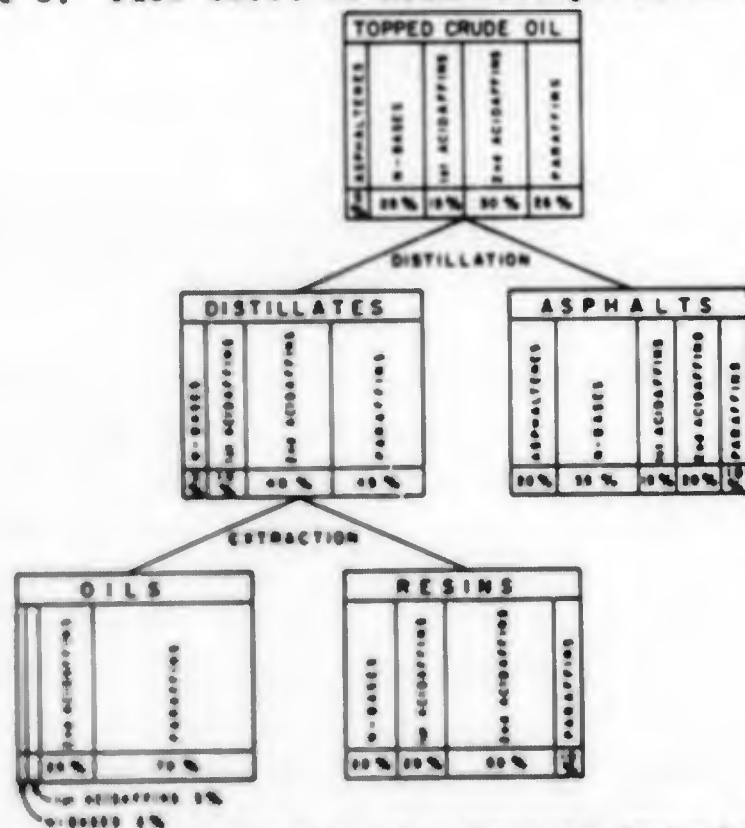


Figure 4. Flow Diagram of a Typical Petroleum Refining Operation in Terms of the Acid Precipitation Analysis

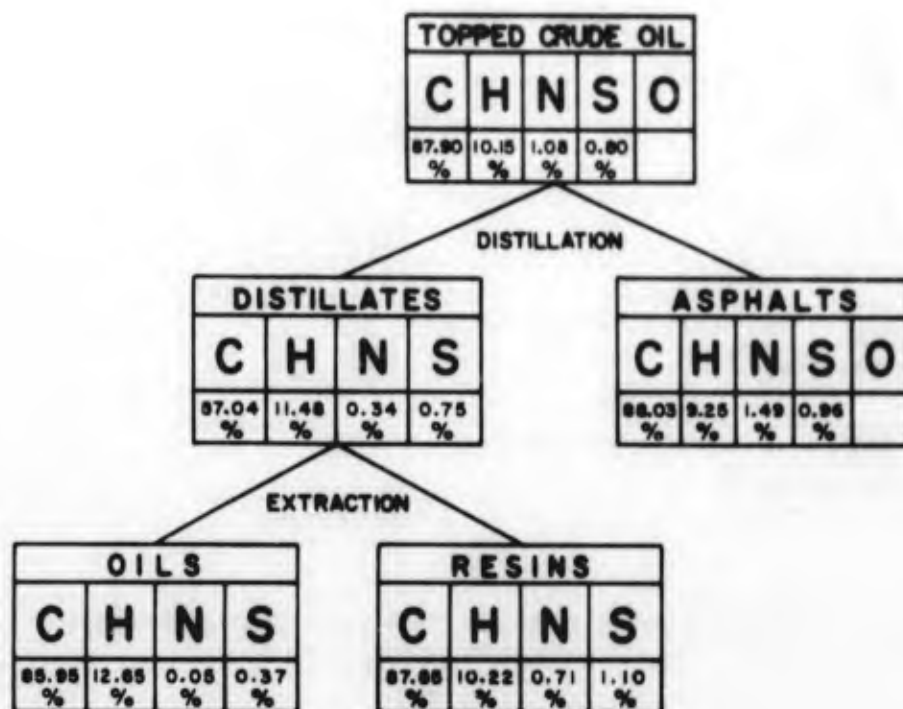


Figure 5. Flow Diagram of a Typical Petroleum Refining Operation in Terms of Elementary Composition

Knowledge of the above facts (summarizing the basic physical and chemical operations performed during the manufacture of paving grade asphalt) is believed to be essential to the understanding of the qualities of bituminous cements used in surfacing. It is also important to realize that asphalt quality depends more on manufacturing procedures than on geographical origin of the crude oil. It is well established that asphalt durability and rheological properties depend on the percentage composition of the asphalt in terms of the fractional components A, N, A<sub>1</sub>, A<sub>2</sub>, and P.

It would be outside the scope of this review to present more than the basic facts of asphalt technology pertinent to this study. Those interested in more details are referred to the

literature references 11, 12, 13, and 14, which are a listing of most of the important publications dealing with the manufacture, chemistry, and physics of asphalt.

## 2. DEFINITION OF CAUSES FOR DETERIORATION OF ASPHALT SURFACES

The two main ingredients of an asphalt pavement are aggregate and asphalt cement. It is the aggregate which provides the strength and load-bearing capacity and the other functional requirements of a pavement pertaining to physical needs. The asphalt cement is quantitatively only a small portion (rarely more than 6 percent) of a pavement. The main function of the asphalt is to cement and hold the aggregate in place during service. The quality of a pavement is thus primarily contingent on the soundness of the aggregate, the quality of sub-base, and type of construction. The mechanism of pavement failures and the causes for main types of distress have been well analyzed and discussed previously (Ref. 15). A chart summarizing the basic facts associated with failures of pavement surfacings has been published (Ref. 16) by the State of California Division of Highways. This chart, reproduced with permission of the California Division of Highways, is shown as figure 6. Proper gradation of the aggregate, composition of the asphalt, correct mixing and adequate design are the primary requirements for good pavement performance.

In evaluating pavement performance and corrective measures to be taken, the fact that the asphalt cement is only one component of the pavement's structure -- and at that, quantitatively only a small one -- must be taken into account. In addition to quality and soundness of the main ingredients, durability of a pavement is dependent on amount and quality of asphalt used, and the voids contained in the structure. The voids of a pavement provide the opportunity for air, water or spilled fuel to enter the pavement and to cause weakening and destruction.

The principal causes of pavement deterioration attributable to the asphalt cement are either insufficient amounts of asphalt or premature embrittlement. In rare cases too much asphalt might

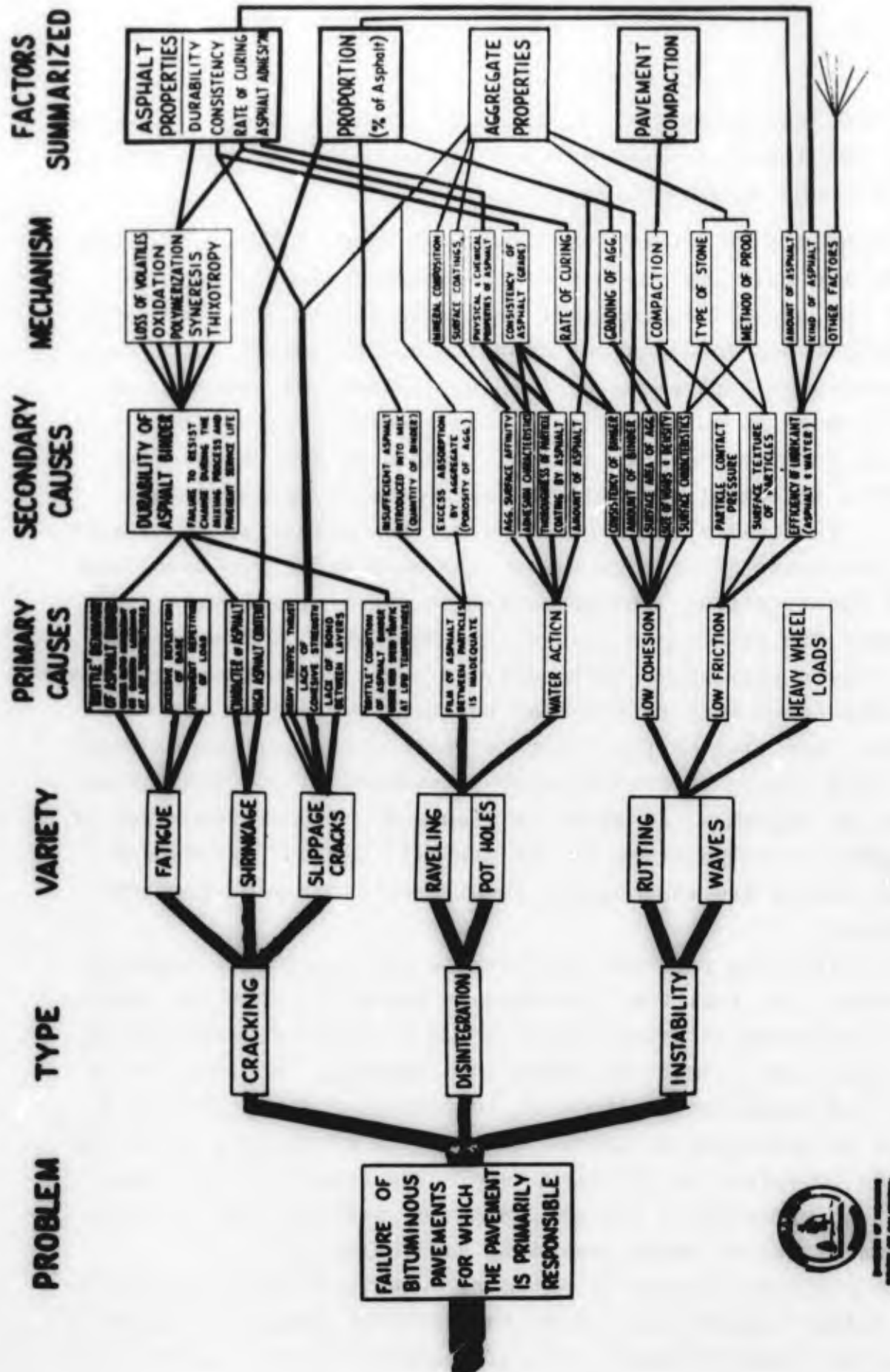


Figure 6. Analysis of Bituminous Pavement Failures  
(Reproduced by permission of California Division of Highways)



have been used causing instability and bleeding. Bleeding can sometimes be corrected by applications of sand or fine aggregate, preferably heated, and rolling. If the amount of asphalt cement used in the construction of the pavement has been so excessive as to cause instability, the pavement must be removed or removed and replaced. Such operations are, however, outside the category of rejuvenation procedures. Insufficient amounts of asphalt in a pavement can be caused by the use of insufficient amounts of binder in the original construction of the pavement or by volatility and thus loss of binder by evaporation.

Previous studies relating the fractional composition of asphalt to embrittlement have well established which components are primarily susceptible to chemical aging (Refs. 5,6,8,9,10). The maltene composition parameter  $(S+A)/(P+A)$  expressive of the ratio of chemically more reactive to less reactive components present in the original asphalt, is a measure of predictable durability.

A durable asphalt designed to meet rheological requirements and having the proper balance of fractional components should have a maltene composition parameter of  $0.8 \pm 0.4$ . Another factor affecting asphalt performance, particularly durability, is the molecular weight of the fractional component asphaltene. All other parameters being equal, the higher the molecular weight of the asphaltene fraction, the better the durability (Refs. 18,19,20, 21). These are, in brief, the fundamental properties of asphalt which permit prediction of performance and life expectancy of a pavement from original properties.

Laboratory tests based on artificially accelerated aging procedures are used to evaluate performance. One such test is the pellet abrasion test employed in the present study. The utility of the pellet abrasion test has been demonstrated in a number of previous studies (Refs. 5,6,9,10,21).

### 3. THE REVERSIBILITY OF THE AGING PROCESS

If pavement deterioration is due to insufficient asphalt, the step to be taken is addition of more asphalt to the pavement. If

is, of course, necessary to select the best asphalt and a suitable preparation and application procedure to accomplish this type of deficiency correction.

The most direct way of adding asphalt to a pavement is to break up the pavement, admix additional asphalt and lay the pavement down again. This process is, however, a major operation and very cumbersome. The most convenient and most desirable way is a spraying operation in which the treating agent penetrates the pavement and combines with the asphalt in place. The difficulty with adding asphalt to a pavement by a spray-on application is that:

- a. Conventional asphalt emulsions do not penetrate pavements to any appreciable depth because these emulsions deposit the asphalt in the uppermost layer of the pavement, plugging the pores and making the pavement impermeable and resistant to deeper treatment, and
- b. Asphalt solutions such as cut-back asphalts, which soften the pavement by means of the solvent, tend to make the pavement unstable and displace the asphalt present in the pavement structure by dissolving the asphalt and washing it off the aggregate.

If pavement deterioration is due to age hardening of the asphalt and not due to insufficient amount of asphalt, the aging process can be reversed by adding components that replace those which, due to chemical changes, became ineffective or insufficient in retaining the cementing properties of the asphalt. In adding the fractional components in which the asphalt is deficient, the asphalt can be replastized and the aging process reversed. In most cases, addition of an asphalt rejuvenating agent free of asphaltenes, i.e., a rejuvenating agent containing only maltenes, can serve the purpose best since a softer binder is usually the objective. Several papers explain the theoretical background for this approach and present data demonstrating the effects obtained (Refs. 7,22). Figure 7 illustrates the effects in terms of restored abrasion resistance. The effectiveness of this kind of



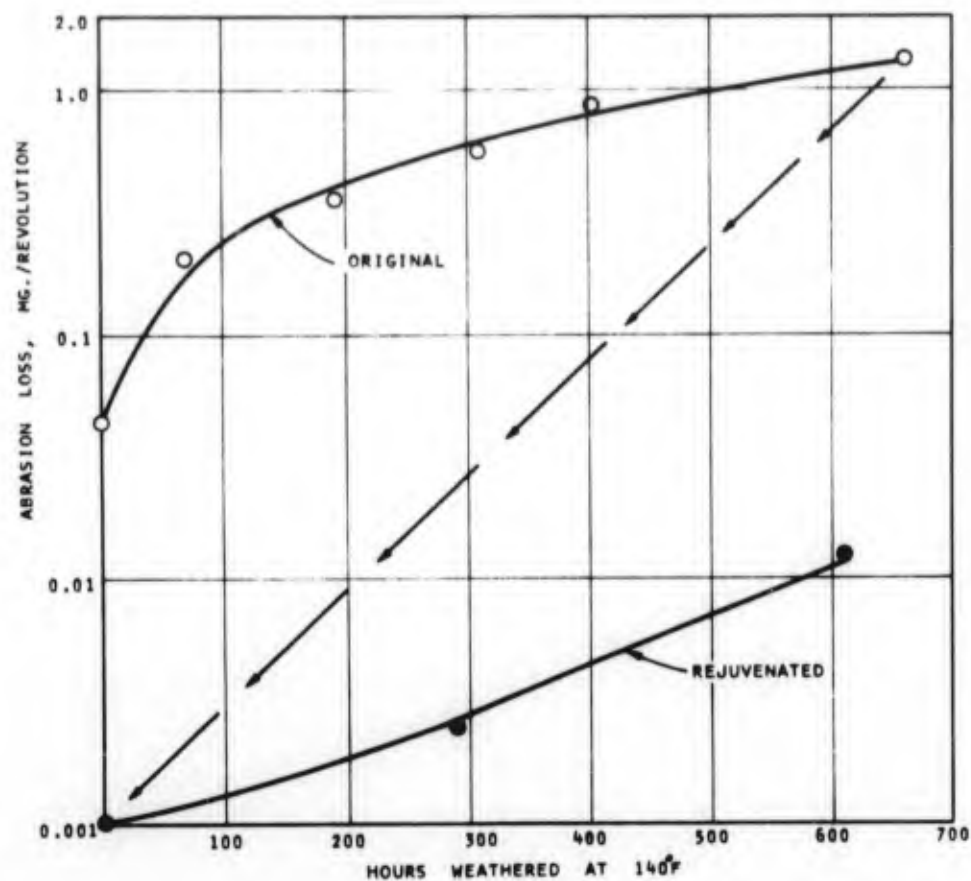


Figure 7. Durability of a 200 to 300 Penetration Asphalt Before and After Rejuvenating.

penetration treatment depends on the composition of the treating agent and its ability to penetrate an aged pavement to the desired depth.

As explained earlier, a durable, serviceable asphalt should have a maltenes composition parameter  $(N+A_1)/(P+A_2)$  in the range of 0.4 to 1.2. Table III shows the relationship between maltenes composition parameter and durability. Since asphalts which deteriorate rapidly are primarily deficient in the components which decrease the maltenes composition ratio, an effective reclaiming agent designed to correct the maltenes composition ratio should contain maltenes with a parameter of about 0.4. This does not apply to rejuvenating agents designed to improve aged asphalts by other means, but asphalt pavement deterioration

Table III

## DURABILITY RATING FOR ASPHALTS OF 60 TO 100 PENETRATION GRADE

Group	$\frac{N+A_1}{P+A_2}$ *	Average Abrasion Loss **		Durability
		%	mg/revolution	
0***	<0.4	--	--	Decreasing durability with decreasing parameter value
I	0.4-1.0	0-10	0.00-0.40	Superior
II	1.0-1.2	5-15	0.20-0.60	Good
III	1.2-1.5	10-40	0.40-1.60	Satisfactory
IV	1.5-1.7	30-60	1.20-2.40	Fair
V	>1.7	>50	>2.00	Inferior

\* Based on original composition.

\*\* Average of abrasion @77°F. (pellet method) before and after aging.

\*\*\* Cheesy and putty-like products of consistency not associated with highway asphalt.

contingent on composition of conventional asphalts can be reversed by addition of certain asphalt constituents.

#### 4. IDENTIFICATION OF ASPHALT CONSTITUENTS CAPABLE OF RESTORING PHYSICAL PROPERTIES

The precipitation method of asphalt analysis defines asphalts as consisting of five basic groups of components identified as the fractional groups of components "A," "N," "A<sub>1</sub>," "A<sub>2</sub>," and "P." The two components "A" and "P" are the most stable fractions in respect to resistance to oxidation. The other components "N," "A<sub>1</sub>," and "A<sub>2</sub>" are subject to oxidation in descending order.

Since in the aging process the amount of fraction "A" always increases at the expense of fractions "N," "A<sub>1</sub>," and "A<sub>2</sub>," the constituents to be added to an asphalt which has age-hardened are these three components if the original composition is to be restored. If further improvement in composition is desired so as to retard deterioration after restoration of the original serviceability, then the reclaiming agent should contain only a minor

portion of fractions "N" and "A<sub>1</sub>" and the replastification should be accomplished by addition of a maltenes-type material high in fractions "A<sub>2</sub>" and "P," having a composition parameter of about 0.4. This is a brief recapitulation of facts pertaining to the individual components of asphalts capable of restoring asphalt properties lost in the aging process.

The restoration of asphalt properties by adding components lost during aging is the most direct way of asphalt rejuvenation. This approach to correcting the effects of aging does not, however, preclude the possibility of using other additives not originally present in asphalt.

#### 5. COMMERCIAL ASPHALT REJUVENATORS

The general effects and utility of asphalt rejuvenators are summarized in the preceding portions of this report. The substantiating facts are described in detail in the literature quoted earlier.

To obtain the information provided by manufacturers of asphalt rejuvenators, twelve companies were contacted by letter, requesting information and samples of the products. The brochures received in reply to this request revealed that the literature on all products manufactured for use as rejuvenators employed to some extent the facts discussed earlier in this paper as supporting evidence for the utility of the products.

Letters were also mailed to State Highway Departments to solicit information as to experience with asphalt rejuvenators. Nearly all highway departments have used some products they consider rejuvenators in their maintenance operations. The opinions expressed as to the utility of the products range from high praise to statements that the products tested were of no avail.

A review of the correspondence and the literature dealing with asphalt rejuvenators shows that all commercially available products recommended can be grouped in the following four categories:

- I     Solutions of asphalts (cut-backs)
- II    Emulsions of asphalts
- III   Emulsions of fractional components of asphalts
- IV    Solutions of ingredients other than fractional components of asphalts believed to be beneficial in asphalt cements.

### SECTION III

#### DESCRIPTION AND DISCUSSION OF LABORATORY WORK PERFORMED

##### 1. LABORATORY TESTS PERFORMED ON PROTOTYPES OF ASPHALT REJUVENATORS

Five products were selected as typical for current products on the market. Two products of category I and one typical for each of the other three categories were tested in order to evaluate the whole gamut of commercially available products. The products were subjected to two series of tests.

Series I - These test procedures were used to identify the products by chemical and physical characteristics and comprised the following determinations:

- a. flash point
- b. viscosity at 77°F.
- c. particle charge
- d. coarseness (sieve test)
- e. storage stability
- f. solids content
- g. consistency of solids (softening point, penetration at 77°F., or viscosity at 140°F.)
- h. chemical composition
- i. compatibility with asphalts

Series II - The tests of this series were performance tests and comprised measurements of:

- a. rate of penetration into asphalt-aggregate mixes
- b. depth of penetration
- c. curing rate
- d. permeability of treated surfaces
- e. change in viscosity of rejuvenated asphalt in aging
- f. ductility of rejuvenated asphalt before and after aging
- g. abrasion resistance of treated asphalt-Ottawa sand mixes

The individual tests are described in Appendixes I through VI.

*The flash point test* indicates the temperature at which a material is easily ignited and thus shows whether or not a fire hazard is involved in its use. Products A and B (category I) flash and burn at or below ordinary ambient temperatures, and should be handled with the same precautions as gasoline. Product C (category II) and product D (category III) are emulsions, containing water, and do not flash. The flash point of product E (category IV) is well above the application temperature recommended by the manufacturer (150° to 160°F.).

*The viscosity at 77°F. test* is a measure of consistency needed to judge the handling characteristics of products. The data measured show that all five products are sufficiently low in viscosity to be pumpable at ordinary temperatures. However, at ordinary temperatures, product E contains some solid material, and must be agitated to keep the solid particles in suspension during transfer operations.

*The particle charge test*, applicable only to the emulsions, shows whether the oil phase of the emulsion has a positive or negative charge. Emulsions with a positive charge (cationic emulsions) preferentially wet the asphalt and not the aggregate. The tests show that product C is anionic and product D is cationic. Care must be taken that tanks and equipment which have been used for an anionic emulsion be carefully and thoroughly cleaned before use with a cationic emulsion, and vice versa. When emulsions of opposite charge are mixed, they coagulate.

*The coarseness (sieve) test* detects emulsions which contain particles coarse enough to clog spray equipment. The test results for products C and D are well within the limits usually included in asphalt emulsion specifications (0.1 percent maximum retained on 20-mesh sieve). The sieve test also serves to separate contaminant particles, which may be examined and identified. The solid particles in product E were separated by filtering through a 100-mesh sieve.



*The storage stability test* consists of keeping specimens in closed containers at 40°F. and 140°F., respectively, for one week and examining for gross separation. No detectable change occurred in products A, C, and D during 7 days' storage at 40°F. and 140°F. Product B contained 0.3 percent of insoluble sediment which settled to the bottom but could be dispersed by agitation. At ordinary temperatures, product E contained 3.5 percent of fine solid particles which dissolved on heating to 140°F. and precipitated on cooling. Since emulsions such as product C and D contain water, satisfactory storage stability at 40°F. does not preclude the possibility of irreversible breaking of the emulsion in storage if not protected from freezing.

*The solids content test* determines the amount and nature of the effective ingredients. The residue from distillation to 680°F., for the solutions (products A, B, and E) or from evaporation of water, for the emulsions (products C and D) indicates the amount of nonvolatile constituents, i.e., those which will remain in the asphalt. Part of the distillate from product E is relatively high-boiling and should therefore be considered a part of the effective residue of this product. The total amount of residue can therefore be considered to be higher than the determined 50.5 percent.

*The consistency tests* (viscosity, penetration, and softening point determination) show whether a hard film or a soft deposit will be left behind after evaporation of the solvent or water. Softening point measured on the residues from products A and B, which have much higher viscosity than normal paving asphalts, indicates that these two products cannot soften an aged pavement (after evaporation of the solvent), and are only an addition to the total asphalt content of the pavement. The residue from product C is an asphalt of paving grade consistency. Product C is, according to the manufacturer, not intended to rejuvenate aged asphalt by replasticization but to increase the asphalt content of pavements which are deficient in asphalt. The residues from products D and E are soft and will soften the aged asphalts to which they are added.

*The chemical composition test* performed on the residue shows, in terms of the five fractional components, whether the rejuvenating agent contains fractions needed to restore the aged asphalt to a favorable composition balance or contains only components which affect consistency physically. The chemical composition indicates that products D and E are high in the components needed to plasticize an aged, embrittled asphalt. Product D, designed to restore or improve original asphalt composition, is high in the nonreactive asphalt components A<sub>2</sub> and P.

*The compatibility tests* measure the miscibility of the effective ingredients with asphalt. The tests performed show that the residues from all five products are compatible with asphalt; product E is less compatible with asphalt than the others, being rated "3" in the filter paper spot test. According to the literature supplied by the manufacturer, product E is compatible with both asphalt and coaltar pitch and can also be used for rejuvenating tar pavements. (Tar pavements were not included in the study.)

*The rate-of-penetration test* performed on briquettes bound with two asphalts known to be different in wettability reveals the speed at which a rejuvenator can be expected to penetrate a pavement surface.

The tests performed showed that Product E penetrated rapidly into briquettes bound with both types of asphalt. Penetration of product D was more rapid than product E in the easily wet briquettes, but slower than product E in the briquettes with poorer wettability. At the typical application rate of 0.2 gsy (gallons per square yard), product A penetrated the easily wet briquettes in less than 4 minutes but took more than 1/2 hour to penetrate the briquettes of poorer wettability. The rate of penetration of product B was much slower; penetration into the hard-to-wet briquettes was incomplete and a film was deposited on the top surface of the briquette. Penetration of product C was



negligible and most of the asphalt was deposited as a thick film on the surface. For all practical purposes, the rate of penetration of product C is negligible.

*The depth-of-penetration test* measures the ability of a treating agent to reach subsurface areas of a pavement and to be absorbed by a pavement.

Products D and E penetrated to a depth of 1/16 to 1/4 inch when applied at 0.2 gsy, and to a depth of about 2/3 inch when applied at 0.8 gsy. Product A penetrated approximately half as deep as products D and E. Product B penetrated approximately 1/16 inch. As mentioned above, penetration of product C was negligible. At the higher application rate of 0.8 gsy, indicative of conditions where puddles may form in depressions of the pavement, penetration was complete only for products D and E. Products A, B, and C deposited heavy films on the surface.

*The curing rate test* was designed to give an indication of time to be allowed before a treated pavement can be open to traffic. At the higher application rate of 0.8 gsy, products A, B, and E made the asphalt briquettes very soft and unstable. This condition lasted only for a few hours with products A and B, which contain solvents that evaporate rapidly. The condition persisted for several weeks with product E, which contains a slower-evaporating solvent. The manufacturer's instructions for application of product B specifies the use of a squeegee, and the manufacturer of product E recommends the use of a drag-broom to redistribute puddled accumulations of the product.

*The permeability test* was designed to test improvements attainable in sealing a pavement through use of an asphalt rejuvenator. Heavy films on the surface make the surface temporarily impermeable, but the surface films soon deteriorate due to wear and exposure. Sealing a pavement to an appreciable depth is a desirable effect of a rejuvenating agent. After removal of the surface films in accordance with the testing procedure, water permeability was found to have been affected very little by product C; however, product B reduced permeability to about three-

quarters of the permeability of untreated briquettes, product D to one-half; product A to one-third, and product E to one-tenth or less. All products were applied to the briquettes' surface at a rate of 0.2 gsy, using the solutions as received, but the emulsions after dilution with one-half their volume of water.

*The change in viscosity of rejuvenated asphalt in aging test* shows, in terms of viscosity at 77°F. of the asphalt extracted from asphalt-sand mixes, to what extent the viscosity of the contained asphalt is changed when a rejuvenating agent is added in amounts of 25 parts of the as-received product per 100 parts asphalt. The viscosity of the asphalt extracted from the aged mixes is an indication of the resistance to hardening with further aging. As predictable from the consistency of the residues, the viscosity measured on the asphalt extracted from treated sand-asphalt mixtures showed that products A and B hardened the asphalt binders, product C softened them only slightly, and products D and E softened them significantly. As predictable from the chemical composition, subsequent hardening with further aging was decreased by product D.

*The ductility of rejuvenated asphalt test* was performed after the asphalt aged to determine the effect of the treating agent in terms of extensibility. The results of the ductility tests did not provide information useful for evaluation of rejuvenating agents. The additive effects of simultaneous change in consistency and composition obscure the picture.

*The pellet abrasion test* measures the resistance of a pellet of asphalt-sand mix to abrasion when tumbled in a rotating square bottle, before aging and after an accelerated oven aging period equivalent to 5 to 10 years field aging. Durability of the binders, measured in the pellet abrasion test, was not significantly improved by treatment with products A and B and was improved only slightly by product C. The durability was improved significantly by products D and E. The low pellet abrasion loss of the aged specimens treated with product D shows the improvement in

resistance to further aging as predictable from the chemical composition.

## 2. DISCUSSIONS OF TEST RESULTS

The results obtained on the five samples of asphalt rejuvenators received from the manufacturers are presented in tables IV and V.

Table IV shows the results of the tests performed to determine the definitive physical and chemical characteristics of the products. The first six columns show the values measured on the products as received. The next columns report the tests performed on the residue, which is the portion of the product remaining after removal of the diluent (solvent or water) and constitutes the portion of the material destined to combine with the asphalt.

Table V shows the results obtained in determining the effects of the products on asphalt. Two sets of specimens designed to determine the interaction of the treating materials with the asphalt mixes were used in these tests. The first set of specimens consisted of asphalt-aggregate briquettes to which the treating agents were applied for the purpose of observing and measuring the ability of the products to enter and combine with a paving mixture. The second set of specimens consisted of asphalt-Ottawa sand mixtures which were tested to determine the degree of improvement attainable after the treating agent has combined with the asphalt. Two different asphalts, typical for medium quality asphalts produced at present, were used. The results of the performance of the untreated asphalts are shown for comparison in judging the effects of the treatment.

Table VI identifies the two asphalts. Both asphalts are of a quality to be expected in asphalts requiring rejuvenation after a relatively short time of service. The main difference between the two asphalts is their resistance to wetting by a treating agent.

Table IV

## PROPERTIES OF ASPHALT REJUVENATING AGENTS TESTED

Product	Flash Point, °C, °F	Visc., 77°F., SFS	Particle Charge	Sieve Test	Storage Stability (4)	Residue %	Tests on Residue			
							Composition %	Visc., 140°F., P	Pen., 77°F.	Soft., Pt., °F
A	<75 (1)	44.0	(3)	-	Stable after 7 days at 40°F. or 140°F.	34.7 (5)	A 44.3	-	2	189
							B 37.1			
							C 4.4			
							D 8.5			
							E 5.7			
B	<75 (1)	137.1	(3)	-	Contains 0.3% sediment	38.0 (5)	B+A 2.92			
							A 38.9	-	8	199
							B 20.8			
							C 12.1			
							D 19.9			
C	(2)	27.7	Negative	0.000% on 20-mesh sieve	Stable after 7 days at 40°F. or 140°F.	60.1 (6)	B+A 1.17			
							A 18.8	1455	75	-
							B 37.5			
							C 13.1			
							D 19.4			
D	(2)	46.9	Positive	Nil on 20-mesh sieve	Stable after 7 days at 40°F. or 140°F.	65.7 (6)	B+A 1.65			
							A 0.5	1.35	(7)	(7)
							B 18.0			
							C 14.0			
							D 40.1			
E	265	17.3	(3)	3.5% on 100-mesh sieve	3.5% solid particles precipitate at 77°F. redissolve at 140°F.	50.5 (5)	B+A 0.47			
							A 13.7	2.43	(7)	(7)
							B 13.5			
							C 29.8			
							D 18.7			
F	265	17.3	(3)	3.5% on 100-mesh sieve	3.5% solid particles precipitate at 77°F. redissolve at 140°F.	50.5 (5)	B+A 2.28			
							A 13.7	2.43	(7)	(7)
							B 13.5			
							C 29.8			
							D 18.7			

(1) Viscosity and continues to burn at 75°F.; minimum flash point not determined. (2) Water emulsion; test not applicable.  
 (3) Solvent system; test not applicable. (4) All the products contain volatiles and must be stored in closed containers; emulsions must be stored above 32°F.  
 (5) Residue by Distillation, ASTM D400. (6) Residue by Evaporation, ASTM D244. (7) Viscous liquid; test not applicable.  
 (8) Synthesis rating under UV light by 2 observers: 1 = severe; 2 = definite; 3 = slight; 4 = trace; 5 = none.

Table V

## PERFORMANCE TESTS

Asphalt Treated Product	Tests on Asphalt-Bound Briquettes										Tests of Ottawa Sand Mixes					
	1		2		3		4		5		6		7		8	
	Rate (time) of Penetration, min		Depth of Penetration, in		Cure Rate, days to loss of half of volatiles		Permeability, 2 min, in/min		Pellet Abrasion Loss		Viscosity, 77°F., 0.05 sec., MP		Microductility, 77°F., mm			
	0.8 gsy	0.2 gsy	0.8 gsy	0.2 gsy			al	in/min	Mixed, unaged \$ mg/rev	Aged 7 days \$ mg/rev	Mixed, Unaged	Aged 7 days	Mixed, Unaged	Aged 7 days		
Untreated Control	V	-	-	-	-	-	22.0	1.51	44.0	1.73	100	5.21	3.9	16.1	53	100
	B	-	-	-	-	-	21.0	1.45	29.7	1.19	100	4.38	6.0	34.5	49	10
A	V	>60	3.75	0.31	0.12	5	7.9	0.55	54.1	2.10	100	4.48	11.8	32.3	110	91
	B	>60	32	0.28	<0.06	1	6.2	0.43	50.7	1.98	100	5.54	12.2	77.0	47	3
B	V	>60	33	0.17	0.06	<1	17.1	1.18	39.2	1.52	95.2	3.77	6.5	28.5	99	101
	B	>60	>60	0.06	0.08	<1	15.0	1.04	36.4	1.46	91.5	4.57	12.0	50.5	33	4
C	V	>60*	>60*	<0.06*	<0.06*	<1	17.8	1.22	13.3	0.53	100	3.52	2.9	14.0	77	88
	B	>60*	>60*	<0.06*	0.06*	<1	21.3	1.46	4.32	0.17	81.2	3.18	4.3	30.5	61	14
D	V	0.87*	0.18*	0.66*	0.20*	2	10.9	0.75	0.17	0.01	1.9	0.08	0.20	0.84	30	43
	B	6.78*	3.58*	0.63*	0.24*	13	10.8	0.74	0.00	0.00	6.2	0.24	0.44	4.1	33	54
E	V	2.08**	0.37**	0.69**	0.25**	>25	2.4	0.7	0.00	0.00	14.3	0.56	0.08	2.5	14	64
	B	2.78**	0.23**	0.69**	0.16**	>25	<0.1	<0.01	0.00	0.00	64.3	2.43	0.12	13.0	22	46

\*2:1 dilution.

\*\*Product applied at 150°F., as recommended by manufacturer..



Table VI  
ASPHALTS USED IN PERFORMANCE TESTS

	<u>Asphalt V*</u>	<u>Asphalt B**</u>
Penetration at 77°F.	58	64
Viscosity, poises at 77°F.	2,070,000	3,500,000
at 140°F.	1870	4922
at 275°F.	2.45	6.89
Composition, % A	10.9	27.8
% N	41.5	26.6
% A <sub>1</sub>	14.2	21.5
% A <sub>2</sub>	20.7	17.8
% P	12.7	6.3
	$\frac{N + A_1}{P + A_2}$	
	1.67	2.00
Durability Group	IV***	V***

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\*Nonwaxy asphalt; "Valley asphalt" from Poso Crude, California (see Ref. 10).

\*\*Waxy asphalt; "Boscan asphalt" from Boscan Crude, Venezuela (see Ref. 10).

\*\*\*See Table III for rating.

## SECTION IV

### INTERPRETATION OF TEST RESULTS

#### 1. EVALUATION OF PRODUCTS TESTED

In evaluating the utility of the products tested on the basis of the laboratory results, the performance tests as summarized in table V are used as the primary yard stick. The data shown in table IV must, however, also be taken into account in specifications since they are of importance for determining ease of handling, safety, logistics, and volume cost.

Additional consideration must also be given to the contemplated mode of application and the specific short-comings of the pavement to be corrected. If, for instance, a spray-on mode of application has been decided upon, only products which penetrate the pavement can be used. If mechanical mixing of a broken-up pavement is specified, penetration is of minor consequence. If the cause of the deterioration is insufficient asphalt, the main consideration should be given to a product which will correct this deficiency without undue softening of the pavement. If the cause of the deterioration is extensive softening of the pavement by fuel spillage, a product should be used which will increase consistency. In consideration of these various factors concerning mode of application and possible types of distress, all types of products tested in this study have some utility in pavement restoration.

In scrutinizing the individual products tested in light of the data presented in tables IV and V, the five products tested as prototypes of commercially available materials can be described as to their potential utility as follows:

- a. Product A is an asphalt treating agent which penetrates and temporarily softens the surface layer of a pavement.

When product A is combined with the asphalt in the pavement, the harder residue remaining after evaporation of the solvent can correct softening caused by fuel spillage. Product A provides a tough upper surface layer well anchored to the pavement. This product could profitably be used for a surface treatment of a pavement rejuvenated by other means. (Handling precautions - consider fire hazard.)

b. Product B is a surface sealing agent which temporarily softens the surface but does not penetrate into the pavement. After evaporation of the solvent, a hard residual surface film is deposited. The product does not improve the durability of an age-hardened asphalt but can be used to fill cracks and correct minor surface blemishes. (Handling precautions - consider fire hazard.)

c. Product C is a non-penetrating sealing material for asphalt-deficient or cracked surfaces not requiring correction of compositional deficiency responsible for hardening. (Handling precautions - keep from freezing.)

d. Product D is a penetrating sealing agent capable of correcting compositional deficiencies responsible for age hardening, replasticing the age-hardened asphalt, and improving resistance to subsequent aging. (Handling precautions - keep from freezing.)

e. Product E is a penetrating sealing agent for asphalt pavements which replastices age-hardened asphalts and reduces water permeability of the pavement. (Handling precautions - heat to 150° to 160°F. and apply at pavement temperatures above 80°F.)

## 2. MANUFACTURERS' RECOMMENDATIONS FOR USE OF INDIVIDUAL PRODUCTS

The manufacturers' literature describing the products tested contain recommendations as to their use. The essence of the recommendations regarding application instructions considered pertinent by manufacturers is as follows:



- a. Product A - Normal application rate 0.10 gsy; sometimes 0.12 to 0.15. Material must be between 65° and 100°F.; ambient temperature above 35°F.; pavement dry and power-swept.
- b. Product B - Application rate 0.1 gsy to 0.5 gsy; apply without heating at temperatures above 33°F.; pavement dry and free of dust and debris; cracks cleaned with compressed air if available; best results obtained after extended period of hot dry weather; squeegee recommended to redistribute excess and to force the material into cracks; 5 lb/yd<sup>2</sup> dust-free sand recommended if treated surface is to be opened to traffic immediately.
- c. Product C - (no instructions from manufacturer.)
- d. Product D - Dilute 2 parts: 1 part water; apply 0.1 to 0.2 gsy at temperatures above freezing; use 1 to 2 lb/yd<sup>2</sup> dry, gritty sand if required.
- e. Product E - Apply 0.05 to 0.20 gsy at temperature of 150° to 160°F. to pavement at 80°F. minimum; pavement should be cleaned of dust and debris; pavement less than 6 months old should be dusted with 0.5 to 1.0 lb/yd<sup>2</sup> portland cement before application of product; after short soaking-in period, brush with drag-broom to remove excess from low areas and deposit it in cracks; apply mineral aggregate if required; rolling recommended for pavements not heavily trafficked.

### 3. RANKING OF PRODUCTS TESTED AS TO THEIR UTILITY AS REJUVENATORS

As pointed out in subsection 1. above, all five prototypes tested can find useful application in the restoration of asphalt pavements. For the purpose of this study, however, a distinction must be made between special utility of individual products in treatment of pavements and the specific utility of a product as a rejuvenating agent. In light of this distinction, the mode of application and extent of reworking a pavement surface is used

in the following discussion as a demarcation line between asphalt rejuvenators and auxiliary products in maintenance operations. Remixing of a pavement surface after it has been broken up is to be considered a salvaging rather than a rejuvenating operation.

Following this reasoning, products B and C must be excluded from further consideration as true asphalt rejuvenators regardless of their potential value in a pavement maintenance operation. Only products capable of penetrating a pavement and combining with the asphalt in place are thus, in the true meaning of the term, to be classified asphalt rejuvenators.

In ranking the remaining three products by merit as to technological utility, the main functions to be judged are:

- a. rate of penetration into a pavement surface
- b. depth of penetration into the pavement
- c. permeability of the treated pavement
- d. durability of the treated pavement

The tests reported in table V determined the performance in accordance with the above-listed desired properties of the rejuvenating agent and rejuvenated pavement. For the purpose of ranking the individual products by numerical values, the test data underlined in columns 1, 2, 4, 5, and 7 (table V) are most significant. The other values should be considered secondary but are useful in expressing preferences.

Table VII shows those values considered indicative of effectiveness for the three products meeting the definition of a rejuvenating agent in that they comply with the two principal requirements of penetrating a pavement and combining with the asphalt in place.

#### 4. CRITERIA FOR PRODUCTS SPECIFICATIONS

The test results obtained and the discussion of the findings suggest that the following characteristics be used in setting up a meaningful product specification for an asphalt rejuvenator:

a. **Product Properties**

- (1) Flash point (minimum)
- (2) Viscosity 77°F. (maximum)
- (3) Sieve test (maximum)
- (4) Storage stability (pass/fail)
- (5) Percent residue (minimum)

b. **Performance Tests**

- (1) Rate (time) of penetration into standard briquette (maximum)
- (2) Depth of penetration into standard briquette (minimum)
- (3) Water permeability of treated briquette (maximum)
- (4) Resistance to embrittlement
  - (a) Abrasion loss of treated asphalt-Ottawa sand mix (maximum); or
  - (b) Change in viscosity during aging (maximum)

The limiting requirements for the above-listed individual tests should be governed by the following:

- a. Fire hazard in storage or handling
- b. Conditions during application
- c. Method of application
- d. Temperatures to be encountered in storage
- e. Cost of effective ingredients
- f. Time between treatment and use of treated area
- g. Required degree of rejuvenation
- h. Required degree of impermeability of treated surface
- i. Anticipated service life of rejuvenated pavement

Table VIII shows suggested tentative specifications based on the results obtained to date.

Table VII

## SELECTED PERFORMANCE CHARACTERISTICS OF ASPHALT REJUVENATORS

Product	Tests on Asphalt-Bound Briquettes			Tests of Ottawa Sand Mixes, Aged 7 Days		
	Penetration, 0.2 gsy			Pellet Abrasion Loss, mg/rev	Viscosity, 77°F. 0.05 sec <sup>1</sup> , MP	
	Rate (time), min	Depth, in	Permeability, in/min			
Untreated Control	-	-	1.45	4.38	34.5	
A	35	<0.06	0.43	5.54	77.0	
D	3.58*	0.24*	0.74	0.24	4.1	
E	0.23**	0.16**	<0.01	2.43	13.0	

\*2:1 dilution

\*\*Product applied at 150°F., as recommended by manufacturer.

Table VIII  
SUGGESTED TENTATIVE SPECIFICATIONS FOR FIELD TRIAL

TEST	SPECIFICATION LIMIT
Flash Point, COC, °F	200 minimum
Viscosity at 77°F., SFS	150 maximum
Sieve Test, % retained on 100 mesh	0.1 maximum
Storage Stability at 40°F. and 140°F.	7 days minimum
Residue, %	60 minimum
Penetration Time <sup>(*)</sup> , minutes	5 maximum
Depth of Penetration <sup>(*)</sup> , inch	0.15 maximum
Embrittlement <sup>(**)</sup>	
(1) Pellet abrasion, aged aged 7 days, % loss	50 maximum
(2) Viscosity at 77°F., 0.05 sec <sup>-1</sup> , after 7 days aging, MP	10 maximum

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\* 0.2 gsy fluid (emulsions diluted to 40% solids)  
on standard briquette

\*\* Product to meet one of the two tests

## SECTION V

### SUMMARY AND CONCLUSIONS

The work performed in the present study resulted in setting up of tentative specifications for asphalt rejuvenators to be tested in the field.

Materials designed to restore serviceability of deteriorating pavements have been laboratory tested as to their pertinent physical and chemical properties and their effects on asphalt. It was shown that a distinction must be made between products of specific utility in treatment of pavements and true asphalt rejuvenators, which in a spray-on application should penetrate a pavement, combine with the asphalt in place, and revive the asphalt in its function as a cementing agent for aggregates.

Since the present study was primarily designed to determine the fundamental physical and chemical properties of asphalt rejuvenators in relation to their desired performance, the main emphasis was given to use of performance tests which provide information indicative of field performance. The physical and chemical characteristics measured on the products have been shown to be suitable for product identification. Criteria suitable for products specifications have been delineated.



## SECTION VI

### SUGGESTIONS FOR FUTURE STUDIES

In view of the fact that the present study provided all the information needed to define and judge an effective rejuvenating agent, it is suggested that tentative specifications be set up and that manufacturers of rejuvenating agents be requested to submit samples for field testing. After selection of the location of the field tests, the manufacturers should be invited to inspect the area and to submit their recommendations for treatment.

A testing program should then be set up to (1) apply the individual products in accordance with recommendations made by their manufacturers, and (2) apply all products submitted by one method and in amounts to be the same for all products to be tested.

The treated areas should be inspected from time to time for appearance. The areas should also be cured immediately after treatment, three months after treatment, and one year after treatment. Comparative values for rate of penetration in the field should be obtained during application of the products. The laboratory tests to be performed on the curves should basically aim at determining the effects obtained in terms of the tests shown in table VII for ranking the products. The rejuvenators submitted should be tested in the laboratory before carrying out the field testing program to determine their compliance with the tentative specifications set up.

After completion of the field tests performed to verify the findings of the study, final, and if needed, revised, specifications for asphalt rejuvenators should be formulated.

Although the performance tests used in this study can be duplicated in research laboratories, it appears advisable to develop simpler tests which can be easily performed in control

and inspection laboratories. It is, therefore, suggested that the tentative specification proposed be used only in securing samples for the proposed field trials and then replaced by purchasing specifications to be developed with the specific aims of simplicity and reliability. These simpler field performance tests should, however, be fully correlated with the performance tests used in the present study.

The proposed field trials, together with the tests on the products to be used in these trials, should provide the information needed to formulate the simplified purchasing specifications and standardized laboratory testing procedures for acceptance specifications.

In order to collect sufficiently extensive data resulting in the setting up of purchasing specifications assuring desired performance without undue restriction of manufacturers, it is suggested that a minimum of five different products be tested. The field tests should be performed on pavement of uniform quality. The test plots need not exceed 100 sq. ft. The laboratory tests for products properties should be the same as used for this study.

The performance tests could profitably be expanded to include tests for skid resistance, resistance to fuel spillage, etc.

APPENDIX I  
TEST METHODS

1. TESTS ON PRODUCTS AS RECEIVED

- a. Flash point, Cleveland Open Cup: ASTM Designation D92
- b. Viscosity at 77°F., Saybolt Furol:  
ASTM Designation D88 (for oils and solutions);  
ASTM Designation D244 (for emulsions)
- c. Particle charge of emulsions: ASTM Designation D244
- d. Sieve Test: ASTM Designation D244
- e. Storage Stability: Specimens are stored at 40°F. and 140°F. for 7 days in closed 8 oz. glass bottles and examined for separation of any phase not readily dispersed.
- f. Residue, percent: ASTM Designation D402 (for oils);  
ASTM Designation D244 (for emulsions).

2. TESTS ON RESIDUE

- a. Consistency Tests:
  - (1) Viscosity at 140°F.: ASTM Designation D2170,  
ASTM Designation D2171.
  - (2) Penetration at 77°F.: ASTM Designation D5, 100 g.  
load, 5 seconds.
  - (3) Softening point, ring and ball: ASTM Designation D36.
- b. Composition, Rostler-White: Proceedings of the Association of Asphalt Paving Technologists, Vol. 31, pp. 72-79.
- c. Compatibility with Asphalt: See Appendix II.

3. PERFORMANCE TESTS

- a. and b. Rate and depth of penetration: See Appendix III and IV.

- c. Rate of Curing: See Appendix V.
- d. Permeability of treated surfaces: See Appendix VI.
- e. Viscosity at 77°F.: California Division of Highways Method 348. (Sliding-plate Microviscometer).
- f. Microductility at 77°F.: California Division of Highways Method 349. To recover asphalt specimens for the viscosity and microductility tests, the asphalt-sand mixes are extracted with benzene, and the benzene solutions are centrifuged to remove sediment, then dried on glass plates in a stream of nitrogen at 77°F.
- g. Pellet Abrasion Test: ASTM Special Technical Publication 277, pp. 64-88, and Proceedings of the Association of Asphalt Paving Technologists, Vol. 35, pp. 123-126. Portions of asphalt-sand mix are mixed with the treating agents and cured overnight before testing.

APPENDIX II  
COMPATIBILITY TESTS

1. MICROSCOPE SLIDE SEGREGATION TEST

a. Scope - When the components of an asphalt or a blend of asphalt components are not mutually compatible, this incompatibility or separation can be observed in a thin film under the microscope. The procedure described provides a method of preparing such a film for observation.

The method is semi-quantitative, since neither the amount nor film thickness of the deposit is exactly controlled.

b. Apparatus -

- (1) Hot plate (Temco HP-A-19.5 B)
- (2) Microscope slides
- (3) Micropipettes, disposable, 100 microliter size (Drummond "Microcaps")
- (4) Microscope, 100X to 400X magnification

c. Procedure - Using a micropipette, transfer to a microscope slide 15 to 20 microliters of a 4 percent solution in benzene of asphalt or asphalt components to be tested. Tilt the slide to form a film 5 to 10 mm in diameter, and let dry. Heat the dried film at 150°C. on the hot plate for 5 minutes, let cool, and examine the film at 100x magnification using transmitted light.

If a clear film is observed, with no structure or network of segregated phases, report a negative result.

Report as positive any widespread irregularity of appearance, which may be a coarse or fine pattern in the film. A dull surface on the film when viewed under

reflected light is observed with the naked eye when severe segregation has occurred.

In borderline cases, re-examine daily for 1 week, using 400X magnification if needed.

## 2. FILTER PAPER SPOT TEST FOR ASPHALT COMPATIBILITY

a. Scope - This procedure provides a means of observing syneresis in an asphalt or blend of asphalt components in the presence of an interfering substrate which increases the tendency of the blend to separate into phases. The material is applied to the paper in solution and the solvent evaporated by applying heat from the periphery of the spot to avoid chromatographic separation from the solution. A drop of an asphalt blend of mutually compatible components will spread evenly with heating on a filter paper, without any type of striation. Where incompatibility of the components exists, there occurs either a flocculation of material on top of the spot, or exudation of a separated oily phase around the periphery of the spot. Flocculation is visible under ordinary light. Exudation of oil becomes visible when viewed under ultraviolet light, because of the strong fluorescence of the acidaffins fractions present in small amounts in the exudate.

The method is semi-quantitative in that neither the amount of material applied to the paper nor the size of the spot is exactly controlled. In order to minimize the subjective element in rating the degree of syneresis, five standard spots judged to represent five rankings are used for comparison with the test spots, and the test spots are rated independently by three observers and their ratings averaged.

### b. Apparatus -

- (1) Filter paper, #1 Whatman, 11 cm diameter
- (2) Pipette, 1 ml, graduated in 0.01 ml
- (3) Rubber bulb and screw clamp
- (4) Ring stand with small clamp



- (5) Ring oven (Nurnberg Scientific, Model RO-31)
- (6) Hot plate (Temco HP-A-1915B)
- (7) Asbestos plates, two, 6 in. x 6 in. x  $\frac{1}{2}$  in.,  
with 2 in. diameter holes in center
- (8) Ultraviolet lamp, 15-watt fluorescent type,  
with reflector
- (9) Filter paper spot test ranking chart (see Table IX)






c. Procedure - To prepare the spot, adjust the ring oven to 170°C. with the 5/8-inch diameter round rings in place. Adjust the vacuum tube beneath the lower ring 1/16-inch below the top surface of the ring. Keep the two rings in contact before and between tests to maintain constant temperature. Adjust the vacuum source to draw a slow stream of air into the tube, leaving the shutter hole opposite the thermometer open.

One to 2 minutes before starting test, center a circle of filter paper between the rings. Using the rubber bulb on the 1-ml pipette, draw 0.2 to 0.3 ml of 4 percent benzene solution of asphalt or blended asphalt components up into the graduated portion of the pipette. Clamp the pipette over the center of the paper with its tip close to the paper surface. Using the screw clamp, gently compress the rubber bulb to deliver 0.1 ml of solution onto the paper in a slow stream. Let the spot dry 1 minute.

Remove the paper containing the dried spot of asphalt and place paper between the two asbestos blocks, centering the spot in the 2-inch opening. Heat the assembly for 10 minutes on a hot plate using 150°C. hot plate surface temperature.

Rate the spots from independent evaluations of three observers comparing the peripheral rings under ultraviolet light with those of the standard spots and observing presence or absence of flocculation under ordinary light.

Table IX  
Filter Paper Spot Test Ranking

Rating	Syneresis	Example
1	Severe	
2	Definite	
3	Slight	
4	Trace	
5	None	

NOTE: In judging syneresis, only the ring surrounding the asphalt deposit is considered. The faint outer ring is a residue from the benzene solution and is disregarded. The ring surrounding the asphalt deposit visible in example 5 is not detectable with the naked eye. It is due to the time exposure needed in taking the photograph.

Rate the degree of syneresis by observing the spot under the ultraviolet light and noting the size of the fluorescent ring surrounding it, disregarding the intensity of the fluorescence. Using the following numerical rating, denote degrees of syneresis:

- 1 - Severe syneresis; i.e., broad ring or series of rings surrounding the central spot (ring can usually be observed under ordinary light in specimens which are rated "1.")
- 2 - Definite syneresis; i.e., distinct ring, 1 to 2 mm wide.
- 3 - Slight syneresis; i.e., thin ring, less than 1 mm wide.
- 4 - Trace of syneresis; i.e., very fine, hair-line ring.
- 5 - No syneresis; i.e., no observable ring.

Note presence of flocculation, observed under ordinary light as heavy black material in the center or around the outer portion of the central black spot, and reported by the notation "f" after the numerical rating.

When observers have rated the tests independently, average their numerical ratings. Write an "f," indicating flocculation, after the rating average if flocculation was noted by more than one of the observers.

## APPENDIX III

### METHOD FOR PREPARATION OF SAND/ASPHALT BRIQUETTES

#### 1. SCOPE

This method describes a procedure for preparation of briquettes of asphalt, graded Ottawa sand, and portland cement filler. The briquettes are used for tests of pavement-treating agents (see Appendix IV, V, and VI).

#### 2. APPARATUS

- a. Mixer, Hobart Kitchenaid, with Pyrex bowl.
- b. Heating mantle, Glas-Col, top mantle for 5-liter, 3-neck flask with one side closed by lace or zipper.
- c. Two adjustable transformers, 7 ½ amps.
- d. Laboratory balance, capacity at least 1610 g, accurate to 0.1 g.
- e. 1-quart mason jar with lid.
- f. Stopwatch.
- g. Oven, with mechanical circulation of air, capable of holding a temperature of  $325 \pm 3^\circ\text{F}$ . (Precision Thelco Oven, Model 18A).
- h. Flexible steel spatula with wooden handle.
- i. Aluminum or tin-plated steel baking pan, 9 x 13 x 2 in.
- j. Carver Laboratory Press.
- k. Mold for molding briquettes 2.5-in. diameter, ca. 1.2-in. high with 2.0-in. diameter 0.318 in. deep reservoir at top, and accessories for removing briquettes from mold, consisting of:

- (1) Mold shell, cylindrical, open at both ends,  
machined from 2  $\frac{1}{2}$ -in. (nominal) i.d. schedule  
80 pipe,  
length 4.00 inches  
diameter, inside 2.50 inches  
diameter outside approx. 2-7/8 inches
- (2) Base plate, 4-in. x 4-in. x  $\frac{1}{2}$ -in. (nominal), with  
cylindrical recess to receive mold shell,  
 $\frac{1}{8}$ -in. deep (nominal).
- (3) Ram (cylindrical, solid, with truncated cone at one  
end),  
diameter 2.49 inches  
length of 2.49-in. diameter portion 4.00 inches  
length of truncated cone 0.318 inch ( $\approx \frac{1}{3}$ )  
diameter, base of truncated cone 2.06 inches  
diameter, end of truncated cone 2.00 inches
- (4) Spacers, 2 inch (nominal)

### 3. MATERIALS

- a. Graded Ottawa sand, ASTM Designation C-109.
- b. Portland cement.
- c. Asphalt (penetration as specified).

### 4. CALIBRATION

Determine the voltage required for the heating mantle to maintain a temperature of  $325 \pm 3^\circ\text{F}$ . in 100 g of graded Ottawa sand (preheated to  $325^\circ\text{F}$ .) in the bowl of the mixer while stirring at speed setting 1.

### 5. PROCEDURE

- a. Weigh  $900 \pm 0.2$  g graded Ottawa sand and  $100 \pm 0.1$  g portland cement into 1-quart Mason jar. Mix thoroughly by tumbling.
- b. Weigh  $75 \pm 0.1$  g asphalt into mixer bowl.

- c. Heat sand-cement mixture in 325°F. oven for  $60 \pm 10$  minutes; heat mixer bowl with asphalt in 325°F. oven for  $10 \pm 1$  minute (time heating periods to end simultaneously). Meanwhile, preheat mantle (inverted 5 liter top mantle slightly deformed fits Hobart mixer bowl). Set mixer speed control on 1 and control speed of mixer through variable transformer (to avoid overflow at start).
- d. At end of heating period, remove sand-cement mixture and mixer bowl with asphalt from oven, dump sand-cement mixture into mixer bowl, place bowl in position on mixer inside preheated mantle and start mixer, increasing voltage to 115 volts promptly.
- e. Mix 5 minutes, including one or two stops if required for scraping sides of bowl with flexible spatula. Dump mixture into metal baking pan. One batch makes 5 briquettes; if several batches are made, hold mixture in an oven at 140°F. until last batch is made and blend batches with spatula.
- f. Cool mixture to approximately room temperature, weigh out 210 g portions and mold briquettes using 10,000 lbs. load ( $\approx 3000$  psi on briquette) for 1 minute.
- g. Rotate the mold to loosen the base plate, and remove it. Rotate the ram to loosen it from the end of the briquette, invert the mold and ram assembly, and, using spacers, press the mold shell down over the ram until the briquette is extruded from the mold. Lift the briquette off the end of the ram, using care to avoid damage to the molded rim.

#### 6. CALCULATIONS REGARDING CAPACITY OF CAVITY IN MOLDED BRIQUETTE

Area of bottom of cavity (end of ram cone)	$\approx 12.2$
Volume of cavity	$1 \text{ in.}^3 = 16.4 \text{ cm}^3$
Area of side of cavity	$2 \text{ in.}^2$



Volume of Liquid in Cavity cm <sup>3</sup>	Wetted Surface in. <sup>2</sup>	Rate Equivalent gal./yd. <sup>3</sup>
1	3.27	0.10
2	3.39	0.20
3	3.52	0.29
4	3.64	0.38
5	3.77	0.46
6	3.89	0.53
7	4.02	0.59
8	4.14	0.66
9	4.27	0.72
10	4.39	0.78
11	4.52	0.84
12	4.64	0.89
13	4.77	0.94
14	4.89	0.98
15	5.02	1.02
16	5.14	1.06

## APPENDIX IV

### METHOD OF TEST FOR RATE AND DEPTH OF PENETRATION OF PAVEMENT-TREATING AGENTS INTO ASPHALT BRIQUETTES

#### 1. SCOPE

This method describes a procedure for determining the rate of penetration of liquids into briquettes made of asphalt, graded Ottawa sand, and portland cement filler. An indication of the manner in which the asphalt is wet by the liquid, and the depth of penetration, may be obtained by examination, under suitable illumination, of the broken surfaces exposed by splitting the treated briquettes. The method for preparation of the briquette is detailed in Appendix III.

#### 2. PREPARATION OF BRIQUETTES

Prepare briquettes according to the method given as Appendix III, using 60 to 70 penetration grade asphalt produced as a residuum of the steam or vacuum distillation of non-waxy crude oil and also using 60 to 70 penetration grade asphalt produced as a residuum of the steam or vacuum distillation of waxy crude oil. Identify the briquettes according to the asphalt contained, as "V" or "B." Briquettes should be molded the same day they are used. The asphalts should substantially match those shown in Table VI.

#### 3. PROCEDURE

Pour or weigh 10 ml (or other specified amount) of the liquid being tested into the reservoir of a briquette and determine the time required for penetration of all of the liquid into the briquette.

After penetration of the liquid into the briquette (or after other specified time interval such as drying or curing period) split the specimen by pressing into one side of the briquette the sharp

edge of an angle iron, to start a crack, then pulling the two halves of the specimen apart manually.

Examine the broken surfaces of the split briquette under suitable illumination, such as ultra-violet light, and measure depth of penetration at several points to note the variation in depth of penetration, if any.

Record identification of briquette, liquid applied, time for penetration, depth of penetration, and other pertinent observations, such as insufficient plating out (liquid running out the bottom) or lateral migration (liquid exuding from sides of the briquette).

## APPENDIX V

### METHOD OF TEST FOR RATE OF CURING OF PAVEMENT TREATING AGENTS

#### 1. SCOPE

This method describes a procedure for determining the relative rate of curing of pavement-treating agents which cure by evaporation of volatile constituents. Since the end point for full cure is often indefinite due to the slow rate of evaporation of the last traces of volatiles, the time for loss of one-half of the volatiles is calculated to indicate the relative curing rate. The actual curing rates in field applications can be expected to be influenced by temperature, humidity and wind conditions.

#### 2. PREPARATION OF BRIQUETTES

Prepare briquettes according to the method given in Appendix III, except for using a 155g portion of mix for each briquette. This lower weight assures that treated briquettes are within the capacity of most analytical balances.

#### 3. PROCEDURE

Weigh a briquette to  $\pm 0.001\text{g}$ , on an analytical balance. Apply 2 ml of treating agent evenly to the flat surface of the depression (reservoir) in the briquette, and weigh the treated briquette immediately; check the weight of the treated briquette at frequent intervals (frequency depending on the evaporation rate of the treating agent) until the weight becomes substantially constant. Calculate and report the time required for loss of one-half the volatile constituents.

## APPENDIX VI

### METHOD OF TEST FOR PERMEABILITY OF ASPHALT BRIQUETTES

#### 1. SCOPE

This method describes a procedure for measuring the rate of permeation of a wetting solution into briquettes made of asphalt, graded Ottawa sand, and portland cement filler.

Surface film, if any, is removed mechanically before the test.

#### 2. APPARATUS

- a. Spot facer,  $\frac{3}{4}$ -in. diameter, mounted in a drill press
- b. Buret, 50 ml capacity
- c. Balance, accurate to  $\pm 0.01$  g.
- d. Stopwatch
- e. Wire gauze square, e.g., 4 x 4 in., 16 or 20 mesh.

#### 3. MATERIALS

- a. Nonionic wetting solution, 1 percent, made by dissolving Triton N-101 in distilled water
- b. Paper towels

#### 4. PREPARATION OF BRIQUETTES

- a. Prepare briquettes according to method given in Appendix III.
- b. Treat briquettes with pavement-treating agents according to method given in Appendix IV, or other specified method, and cure for specified time interval.
- c. Using the spot facer mounted in a drill press with a suitable stop, drill a  $\frac{3}{4}$ -in. diameter hole exactly 2 mm deep in the surface of the reservoir of the cured, treated briquette.

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13. ABSTRACT (Distribution Limitation Statement No. 3) The physical and chemical aspects of the manufacture of paving grade asphalts and of asphalt performance are presented in sufficient detail to provide background information for understanding the physical, chemical and mechanical aspects of paving rejuvenation. The principal facts discussed are substantiated by references to the technical literature, which should be consulted for additional information regarding details. Theoretical and practical considerations pertaining to the use of asphalt rejuvenators are discussed, and laboratory methods of evaluating the utility of asphalt rejuvenators are detailed. The present status of knowledge regarding asphalt manufacture, causes of deterioration of asphalt pavements, and reversibility of the deterioration are depicted in graphs and flow charts. Five specimens, typical of commercially available asphalt rejuvenators, and tests performed on them are described. The test results obtained are tabulated. It is recommended that field tests be conducted before establishing standardized laboratory procedures for acceptance specifications for asphalt rejuvenators. Criteria to be used in tentative specifications to be issued before field testing are suggested.			

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